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**APPLICATION OF GENERAL TECHNOLOGY  
APPLICATIONS, INCORPORATED (GTA)  
BLENDING PROCESS TO ANTIMISTING FUEL  
ADDITIVES**

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**U. S. DEPARTMENT OF TRANSPORTATION  
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## Technical Report Documentation Page

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15. Abstract  This program examines the applicability of a proprietary blending process to blending antimisting additives in JET A aviation fuel. The first phase of the program covers use of the process with a high molecular weight polyisobutylene. The second phase covers the use of the process with Imperial Chemical Industries, Ltd's proprietary polymer, FM-9. The results of the program indicate that high molecular weight polyisobutylene can be blended rapidly with JET A using the proprietary process and that the resulting blend demonstrates antimisting behavior within minutes. There was no evidence that the proprietary process is effective in the blending of FM-9 in JET A.			
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## INTRODUCTION

### PURPOSE.

The Federal Aviation Administration (FAA) Antimisting Fuel Program is currently investigating the use of an Imperial Chemical Industries, Ltd., (ICI) proprietary polymer additive designed to increase the antimisting characteristics of typical jet engine fuels. General Technology Applications, Inc., (GTA) has a proprietary process for rapidly dissolving high molecular weight polymers, such as those known to have antimisting qualities in hydrocarbon liquids.\* Application of the GTA blending process to the FAA antimisting program could lead to the solution of a number of operational problems which have been identified in the use, blending and degrading of antimisting additives.

### BACKGROUND.

The FAA Antimisting Fuel Program has been in progress for approximately three years. Recent work by the FAA has identified fuels in which the misting properties have been decreased by addition of relatively low concentrations (e.g., 3000 ppm) of very high molecular weight polymers. The FAA's Engineering and Development Program Plan for Antimisting Fuel defines the critical issues facing commercialization of antimisting fuels.(1) They are:

- "1. Can the modified fuel be made available in adequate quantities and at an acceptable cost with adequate control for worldwide deployment?
2. What degree of protection would the antimisting fuel provide in post-crash fuel fires?
  - a. A few minutes after refueling; i.e., typically in the take-off case.
  - b. At the end of a flight cycle; i.e., typically in the approach/landing case.
3. What changes, if any, are necessary to enable a civil aircraft to use the antimisting fuel? For example, are changes necessary to fuel system tanks, pumps, filters, etc., and/or to the engine fuel management system?
4. Is it feasible in day-to-day commercial operation to blend the fuel at the fueling point?

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\* A patent application covering this process was filed by GTA in February 1979.

5. Must the antimisting fuel be degraded (subjected to some mechanical shearing or other process) before it is suitable for operation in an engine system, and if so, how can this controlled degradation be achieved?"

GTA, in connection with experimental work in the drag reduction field, has developed a process for rapidly (a few seconds) dissolving very high molecular weight polymers in liquid hydrocarbons with minimal degradation of the polymer. A potentially important benefit to the antimisting program would be the ability to examine a wide range of high molecular weight polymers which might otherwise have been dismissed from consideration for reasons relating to cost and difficulty of blending with JET A fuel.

The GTA blending process utilizes cryofracturing to produce active surfaces on polymer particles which interact with a liquid hydrocarbon solvent accelerating the rate at which solutions are formed. The rate at which a polymer dissolves in a given solvent generally increases greatly when the proprietary GTA process is used over conventional processes. The GTA process both subdivides and activates surfaces of polymers in a colesive solid state, ensuring rapid progress toward thermodynamic equilibrium between dissolved polymer and solvent molecules. Thermodynamically, polyisobutylene is an ideal solute for JET A and the GTA solution process is achieved rapidly.

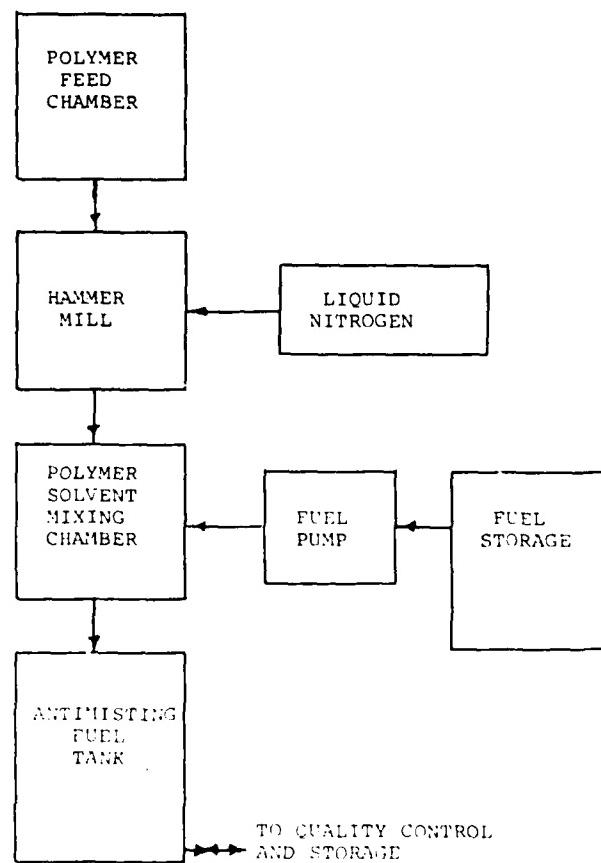
The compatibility of polyisobutylene and an alkane rich solvent like JET A experimentally reflected in a viscosity which is a simple essentially temperature independent, function of concentration. The viscosity of solutions of PIB in a thermodynamically favorable solvent is a function of the following type, at a constant temperature, T

$$\eta = \eta_s (1 + \eta) c + kc^2 + \dots$$

where  $\eta$  is the viscosity of the solution at polymer concentration  $c$ ,  $\eta_s$  is the viscosity of the solvent,  $(\eta)$  is the intrinsic viscosity and  $k$  is the Huggin's constant. In the case of a solvent and polymer that are intrinsically compatible, the  $(\eta)$  and  $k$  terms are essentially independent of temperature over a very broad range. When the compatibility is poor,  $(\eta)$  and  $k$  are functions of a temperature with  $(\eta)$  generally decreasing with decreasing temperature down to the Theta temperature, at which point the solution is thermodynamically unstable and a colloid or gel phase is observed. The GTA process is not effective at temperatures below the Theta point.

DESCRIPTION OF THE GTA BLENDING PROCESS. The system used consists of a hammer mill cooled by liquid nitrogen, a variable flow positive displacement pump for the JET A fuel, a fuel spray tube and housing for simultaneous mixing of the fuel and cryofractured polymer at controlled rates and a collection tank with a spigot (Figure 1). The flow of fuel into the spray tube is coordinated with the cryofracturing of the polymer to produce the desired concentration of polymer in JET A fuel. The pumping rate can be varied from 0.5 to 5 gallons per minute. Thus 5 gallons of 0.3 percent concentration of polymer in JET A can be

FIGURE 1  
BLOCK DIAGRAM OF CRYOFRACTURING  
POLYMER SOLUTION PROCESS



produced in about 1 minute.

While investigation of the operational feasibility of the GTA process is not a part of the current contract it is important to the understanding of the potential application of the GTA process in the antimisting program to know that polymer collected from the cryofracturing mill can be preserved at cryogenic temperatures in a cryogenic container until it is introduced into a hydrocarbon liquid without losing its rapid blending characteristics.

## DISCUSSION

### GENERAL.

The FAA chose two polymers to be blended with JET A by GTA using its proprietary process. The two polymers chosen were BASF's Opanol B-230, a high molecular weight polyisobutylene, and ICI's FM-9. The test program was divided into two phases, the first phase dealing with Opanol B-230, and the second phase with FM-9. Polyisobutylene was selected for demonstrating the GTA blending process because it is representative of a class of hydrocarbon additives that could impart antimisting behavior to JET A. It is one of the amorphous polyolefins that exhibit elastomeric properties, particularly at the higher molecular weights. It was anticipated that Opanol B-230 would provide a good test of the GTA blending process because of its very high molecular weight (Figure 2). It was also anticipated that the ratio of elastic properties to viscosity at a given concentration would be higher than that exhibited in past studies based on lower molecular weight polyisobutylenes. This would be a consequence of the fact that as the molecular weight is increased the concentration required for a given level of antimisting behavior is decreased.

The FAA provided a team to conduct quality control and screening tests of the antimisting behavior of GTA prepared samples of antimisting fuel blends. The tests were designed to determine (1) how quickly antimisting effectiveness was obtained, (2) the degree of antimisting protection provided and (3) the specifications of the resulting fuel. The test methods used for the antimisting evaluations were the Inertial Rheometric Pump and the FAA's Flammability Comparison Test Apparatus. Standard fuel specification tests were conducted.

GTA provided backup quality control and characterization test procedures at its facility. A Brookfield, "Synchro-Lectric" viscometer, model LVT with a 0.75 inch diameter by 3 inch long cylindrical bob was used to quickly determine concentrations of polymer in antimisting fuel solutions produced by the GTA system. The calibration curve relating the Brookfield viscosity reading to concentration is shown in Figure 3. This calibration curve was based on determining concentrations by a gravimetric procedure. The polymer in a test solution was quantitatively precipitated out of JET A by adding acetone while stirring the mixture. The precipitated polymer was removed, washed

FIGURE 2

RELATIONSHIP BETWEEN OPPANOL GRADE NUMBER  
AND VISCOSITY AVERAGE MOLECULAR WEIGHT

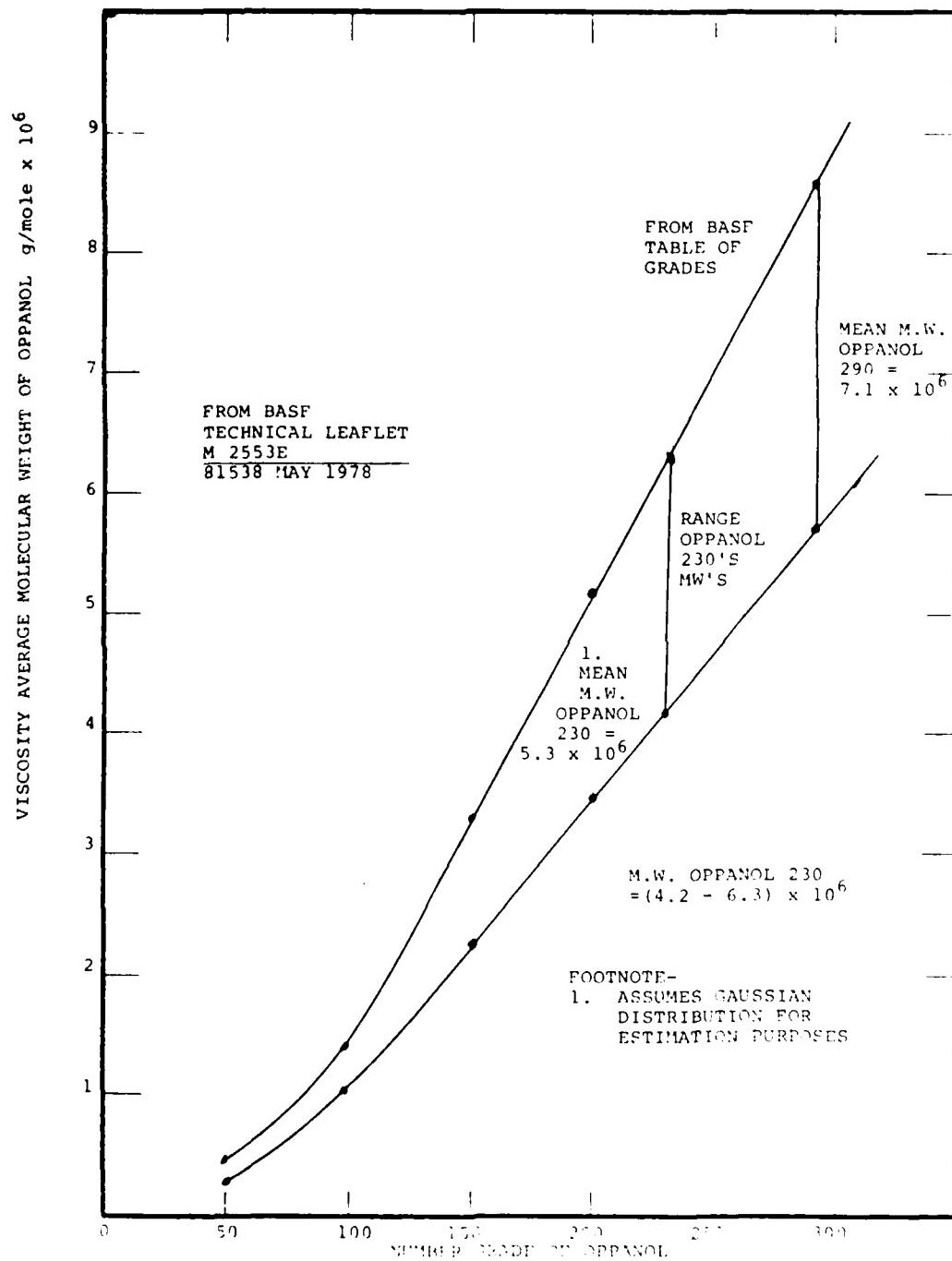
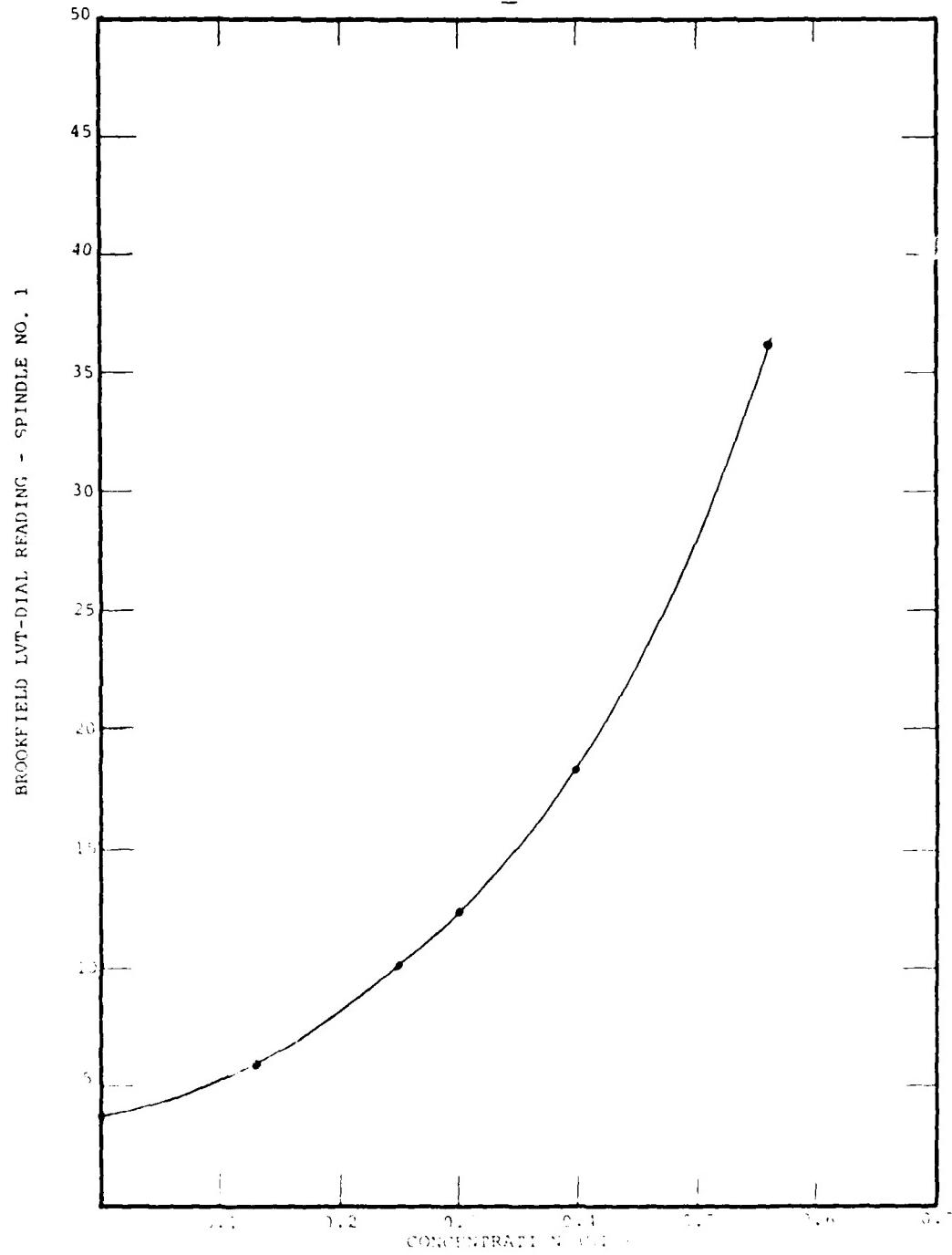


FIGURE 3

CALIBRATION CURVE RELATION VISCOSITY TO  
CONCENTRATION OF B-230 IN JET A FUEL AT  
 $t = 20^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$



three times with 100 ml addition of acetone and vacuum dried to constant weight (under vacuum at 50°C).

This procedure, used to establish the calibration curve for the Brookfield viscometer, was also used to measure the concentrations of polymer in each sample of Oppanol B-230/JET A fuel supplied to the FAA. This procedure provides a more accurate measure of concentration than the viscometer concentrations. The vacuum drying operation of the gravimetric procedure requires approximately 14 hours to complete. The vacuum drying procedure and apparatus is illustrated in Figure 4.

The fuel spray tester shown in Figure 5 was used to screen antimisting behavior of prepared samples within a few minutes after completing the blending operation. This tester applies a pulsating pressure wave to a stream of liquid emerging from a 0.01 inch diameter orifice in a sapphire plate. Antimisting behavior relative to JET A or another reference sample of fuel can be observed directly.

#### PHASE I.

INITIAL BLENDING TEST. On October 8, 1980, a 10 gallon sample of Oppanol B-230 in JET A was prepared at the GTA facility in Gaithersburg, Maryland. The sample was delivered to the FAA Technical Center, Atlantic City, New Jersey on October 9, 1980. The concentration of B-230 was measured initially to be 0.3 percent using the viscosity-concentration calibration curve (Figure 3). Subsequent gravimetric analysis showed the concentration to be 0.285 percent. Fuel specification tests were run on the 0.285 percent sample of October 10, 1980 at the FAA Technical Center. Summary FAA findings were:

<u>TEST</u>	<u>RESULTS</u>
a. Water shedding	Equal to JET A
b. Water interface	Equal to JET A
c. Gum	4000 ppm
d. Viscosity at 20°C	25 to 30 cps

The first antimisting tests were run on the 0.285 percent sample on the same day. The Inertial Rheometric Pump indicated that the 0.285 percent B-230/JET A fuel would demonstrate significant antimisting behavior. The same sample passed the complete range of tests conducted on the Flammability Comparison Test Apparatus.

Upon completion of the first series of tests on October 10, 1980, FAA directed GTA to prepare the remaining 65 gallons of polymer samples called for under the contract using Oppanol B-230. The preparation and delivery schedule was as follows.

FIGURE 4

VACUUM DRYING APPARATUS USED IN  
GRAVIMETRIC PROCEDURE FOR DETER-  
MINING POLYMER CONCENTRATION

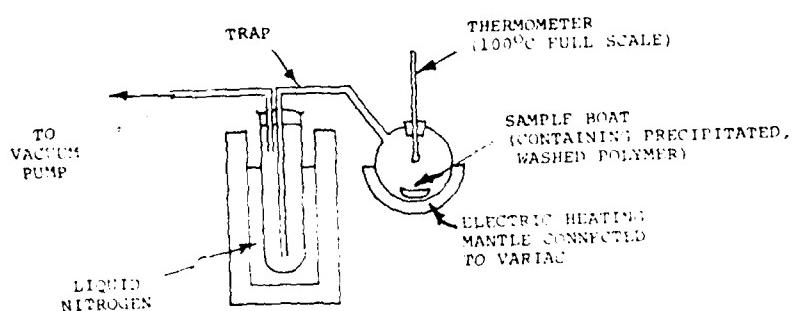
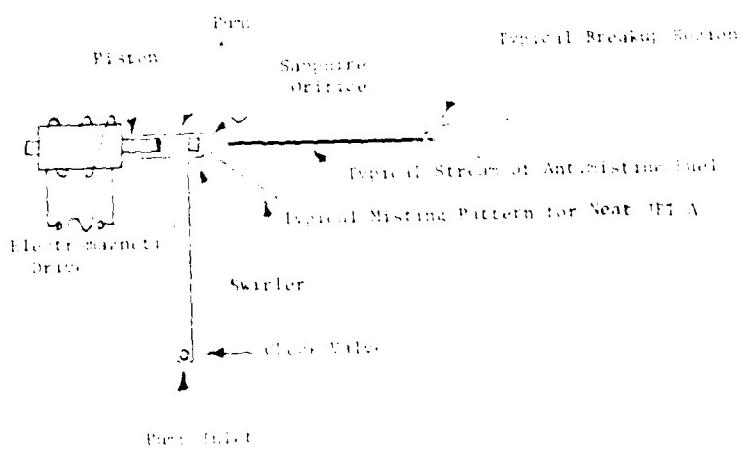


FIGURE 5

BASIC PRINCIPLE OF DIP-TYPE  
SPRAY TESTER FOR SCREENING  
ANTIMISTING SOLUTIONS



<u>QUANTITY (GALLONS)</u>	<u>CONCENTRATION ORDERED BY FAA (WT%)</u>	<u>CONCENTRATION DELIVERED</u>	<u>PREPARATION DATE</u>	<u>DELIVERY DATE</u>
15	0.10	0.100	10/14/80	10/18/80
10	.15	.139	10/14/80	10/18/80
10	.15	.139	10/14/80	10/18/80
20	.20	.178	10/14/80	10/18/80
10	.30	.276	10/14/80	10/18/80

FAA OBSERVED BLENDING TEST. A team from the FAA Technical Center visited the GTA facility on October 17, 1980, to conduct tests to determine how rapidly a B-230/JET A blend reached antimisting effectiveness. A portable Inertial Rheometric Pump was set up by the FAA team. A seven gallon sample of a B-230/JET A fuel mix was prepared using the GTA blending system. The sample was prepared in approximately 2 minutes.

Immediately upon completion of the blending operation a sample was taken for the Inertial Rheometric Pump test, the viscometer concentration check and the quick fuel spray test. The sample had a slightly turbid appearance. A portion of the sample was put under vacuum using a rotary oil vacuum pump. The turbidity disappeared within seconds indicating that the sample had contained trapped gas. The source of the gas was not determined.

After allowing 1 to 2 minutes to warm the sample to 20°C the viscometer calibration curve revealed a concentration of 0.28 percent. The spray test was conducted within 2 to 3 minutes after completion of the polymer-fuel blending operation. It produced a long thin stream of fuel instead of the profuse, fine mist characteristic of JET A. The FAA team using its Inertial Rheometric Pump determined that the antimisting character of the B-230/JET A sample was equivalent to that obtained from the B-230/JET A sample tested at the FAA Technical Center on October 10, 1980.

Subsequent viscometer checks of the concentration of the B-230/JET A sample made at 20 minute intervals over the four hour period following the blending operation provided readings indicating no appreciable change in concentration within the sensitivity of the method.

It is apparent from the blending test conducted for the FAA team that full antimisting effectiveness of the B-230/JET A sample was obtained within 3 to 4 minutes after introducing the polymer into the JET A fuel. The constant viscosity of the B-230/JET A sample from the initial viscometer readings at 3 to 4 minutes after the blending operation until the final reading 4 hours later substantiates this conclusion. The results of the Inertial Rheometric Pump test and the visual

observations of the fuel spray test are additional support.

RESULTS OF THE FLAMMABILITY COMPARISON TEST APPARATUS. A matrix of 73 tests of B-230/JET A samples was conducted at the FAA Technical Center using the Flammability Comparison Test Apparatus (Table 1). A 0.285 percent concentration from the first B-230/JET A sample delivered to the FAA passed all tests, confirming earlier FAA results. Initial tests with 0.139 percent and 0.178 percent samples produced anomalies which could not be accounted for from past test experiences. Procedures were reviewed and a flush cycle was introduced between each test. Subsequent runs resulted in the 0.139 percent sample passing tests through 16 milliliters/second of fuel flow and 70 meters/second of air flow. The 0.178 percent sample passed tests through 18 milliliters/second of fuel flow and 70 meters/second of air flow. The 0.1 percent sample which was tested before the change in test procedure passed the 10 milliliter/second tests at 50, 60 and 70 meters/second air flow and the 12 milliliters/second test at 50 meters/second air flow.

TABLE 1  
FLAMMABILITY COMPARISON TEST APPARATUS

	<u>CONCENTRATION OF B-230 (WT%)</u>	<u>FUEL FLOW (ML/SEC)</u>	<u>AIR FLOW (MTR/SEC)</u>	<u>PASS/FAIL</u>
1.	0.285	10	50	pass
2.	.285	10	60	pass
3.	.285	10	70	pass
4.	.285	12	50	pass
5.	.285	12	60	pass
6.	.285	12	70	pass
7.	.285	14	50	pass
8.	.285	14	60	pass
9.	.285	14	70	pass
10.	.285	16	50	pass
11.	.285	16	60	pass
12.	.285	16	70	pass
13.	.285	18	50	pass
14.	.285	18	60	pass

TABLE 1 (CONTINUED)  
**FLAMMABILITY COMPARISON TEST APPARATUS**

	<u>CONCENTRATION OF B-230 (WT%)</u>	<u>FUEL FLOW (ML/SEC)</u>	<u>AIR FLOW (MTR/SEC)</u>	<u>PASS/FAIL</u>
15.	0.285	18	70	pass
16.	.285	19.5	50	pass
17.	.285	19.5	60	pass
18.	.285	19.5	70	pass
19.	.10	10	50	pass
20.	.10	10	60	marginal
21.	.10	10	60	marginal
22.	.10	10	70	pass
23.	.10	10	55	pass
24.	.10	10	65	marginal
25.	.10	12	50	pass
26.	.10	12	60	fail
27.	.10	12	70	marginal
28.	.15	10	50	pass
29.	.15	10	60	pass
30.	.15	10	70	pass
31.	.15	12	50	pass
32.	.15	12	60	pass
33.	.15	12	70	pass
34.	.15	14	50	fail
35.	.15	14	60	marginal
36.	.15	14	70	pass
37.	.15	16	70	pass

TABLE 1 (CONTINUED)  
**FLAMMABILITY COMPARISON TEST APPARATUS**

	<u>CONCENTRATION OF B-230 (WT%)</u>	<u>FUEL FLOW (ML/SEC)</u>	<u>AIR FLOW (MTR/SEC)</u>	<u>PASS/FAIL</u>
38.	0.15	18	70	fail
39.	.15	16	60	marginal
40.	.15	14	60	marginal
41.	.15	16	60	pass
42.	.15	16	50	pass
43.	.15	14	50	fail
44.	.15	18	50	fail
45.	.20	14	50	pass
46.	.20	14	60	pass
47.	.20	14	70	pass
48.	.20	16	50	marginal
49.	.20	16	60	fail
50.	.20	16	70	fail
51.	.20	18	50	fail
52.	.20	18	60	pass
53.	.20	18	70	fail
54.	.20	19.5	60	fail
55.	.20	19.5	70	fail
56.	.20	18	60	marginal
57.	.20	16	70	incomplete
58.	<u>FLUSH CYCLE INTRODUCED</u>			
59.	.20	16	70	pass
60.	.20	18	70	pass

TABLE 1 (CONTINUED)  
**FLAMMABILITY COMPARISON TEST APPARATUS**

	<u>CONCENTRATION OF B-230 (WT%)</u>	<u>FUEL FLOW (ML/SEC)</u>	<u>AIR FLOW (MTR/SEC)</u>	<u>PASS/FAIL</u>
61.	0.20	18	60	pass
62.	.20	16	60	pass
63.	.20	16	50	pass
64.	.20	18	50	pass
65.	.15	14	50	marginal
66.	.15	14	60	pass
67.	.15	14	70	pass
68.	.15	16	50	fail
69.	.15	16	60	fail
70.	.15	16	70	pass
71.	.15	18	50	marginal
72.	.15	18	60	marginal
73.	.15	18	70	fail

The results of FM-9 tests in the Flammability Comparison Test Apparatus were used as a comparison to determine the relative effectiveness of the B-230/JET A samples. The best result achieved by a 0.3 percent FM-9 fuel was a pass at 16 milliliter/second fuel flow and 60 meters/second of air flow. It is concluded, therefore, that a 0.139 percent B-230/JET A fuel is at least equal in performance in the FAA's Flammability Comparison Test Apparatus to the 0.3 percent FM-9 fuel.

#### PHASE II.

TEST PROGRAM. The FM-9 additive used in the FM-9 antimisting fuel program is in the form of a fine powder. The GTA process requires breaking some intramolecular bonds and therefore, it is necessary that the polymer being blended be in a homogeneous solid state, thereby permitting intramolecular stresses on molecules to exceed bond strengths. Since the manufacturer of FM-9 was unable to supply its additive in a form suitable for this program it was necessary to devise methods for consolidating and fusing the powdered polymer. Three approaches were selected by the FAA to be developed by GTA to

be applied to FM-9 to maximize the potential of its blending process.

1. Pelletizing by pressure (10 tons per square inch for 2 to 3 minutes) under vacuum.
2. Melting under vacuum.
3. Blending a 75 percent concentration of FM-9 in JET A.

GTA also was to develop an approach for working with an FM-9 slurry mix, containing a carrier fluid.

FM-9 is controlled as a proprietary product by its manufacturers. The assumption made in the test program was that the manufacturer would not make FM-9 available except for the actual preparation of FM-9/JET A fuel blends and further that only limited information would be made available with regard to FM-9's chemical and physical properties. The test program, therefore, was to be designed using a substitute polymer to be selected by the contractor using only data found in the open literature.

Pelletizing Under Vacuum. The Royal Aircraft Establishment supplied information to the FAA indicating that FM-9 powder took on the appearance of plexiglas when pelletized by pressure at 10 tons per square inch for 2 to 3 minutes under vacuum.

A hydraulic press capable of exerting 30 tons per square inch on the ram was used to compress powder within a pellet cell with 1 1/8 inch diameter piston and anvil faces. The pellet cell was modified to be used under vacuum. An electric hot air heat gun was used to adjust the temperature of the pellet cell.

A substitute polymer for use in developing solidification and blending processes was selected on the basis of (1) information available in the open literature of the antimisting fuel program and (2) the report that FM-9 powder took on the appearance of plexiglas when subjected to 10 tons per square inch for 2 to 3 minutes in a vacuum cell. The second point of information is consistent with a relatively low softening or melting temperature. Assuming that FM-9 is plasticizer-free, the low softening point could be due to the presence of two mers, i.e., FM-9 would be a co-polymer. (The polarity of FM-9 is alluded to in the literature (reference 2).

To be consistent with the information summarized above, a co-polymer of polyvinylpyrrolidone and vinyl acetate (PVP/VA) was selected for protocol development and testing. It was found, however, that heat had to be applied to the cell during pressing in order to yield a fused pellet resembling plexiglas. Failure to heat the cell resulted in a dense, compressed powder pellet that fractured easily offering minimal opportunity to fracture covalent bonds.

Although the selection of PVP/VA was believed to be consistent with the information available on FM-9, several other polymer powders

were tested in the pelletizing apparatus. These were polyvinyl acetate (PVA), polyethylene oxide (Polyox), polypropylene (PP) and polyvinyl chloride (PVC). As expected none of these yielded pellets resembling plexiglas, even at pressures up to 100 tons per square inch (on a 1/2 inch diameter die, at room temperature (68°F)). PVA, for example, yielded a pellet after treatment at 100 tons per square inch for 2 minutes (vacuum applied) that could be abraded to flake off particles with finger pressure. In summary, GTA did not produce a satisfactory pellet using any of these candidates at a pressure of 10 tons per square inch for 2 to 3 minutes under vacuum at ambient temperatures.

Satisfactory appearance (approaching the plexiglas condition) was approached by 6.3 grams of PVP/VA subjected to 10 tons per square inch (read on scale for a 1 1/4 inch die) and heated for 4 minutes. The outside cell temperature was 62° + 10°C after heating. The cell was kept under pressure for a total elapsed time of 10 minutes. The resulting pellet measured 1.125 inch in diameter by 0.325 inch thick and had a density of 1.19 gram per cubic inch.

Heat rather than pressure, was found to be the most critical requirement of the pelletizing operation. Pellets produced by heat and pressure treatment exhibited a slight asymmetry in the extent of fusion from top to bottom surface. The top of the pellet (facing the movable piston) appeared more resinous and clear than the bottom. The bottom surfaced (facing the anvil) had a more opaque appearance. This slight asymmetry was probably due to a temperature gradient running to lower temperatures at the anvil or the cell which was very close to the large heat sink comprising the base of the hydraulic press.

After receiving the pelletizing test results, PVP/VA was selected for the pelletizing approach to fuel blending. The next step was to reduce the pellets to a size which could be handled by the cryofracturing mill. It was determined that the pellets produced from PVP/VA could be shattered easily with a hammer.

Finally, 50 grams of pellets were produced, shattered and ground in the cryofracturing mill in order to determine mill power and cooling requirements. This information was necessary to determine fuel flow rates for the blending process. It was established that the 94.2 grams required to produce a 10 gallon fuel blend could be cryofractured in 2 to 4 minutes. The flow rate of the fuel was established accordingly (approximately 4 gallons per minute).

Melting Under A Vacuum. No information was made available to contractor with regard to fusion of FM-9 prior to the day scheduled for blending with JET A at the GTA facilities.

A National Appliance vacuum oven with a capability of maintaining a temperature of 200°C was used with a rotary, oil-type vacuum pump. A teflon tray, with integral teflon pillars, was constructed to hold up to 275 grams of polymer (with a density of 1 gram per cubic

inch). The flexible container (10.5 inches x 7.5 inches x 2.5 inches) was designed so that fused polymer could be extracted in a form which could be fractured readily into 3/8 inch square pieces for use with the cryofracturing mill.

In order to test the vacuum fusion and removal process, 5 gram samples of PVP/VA substitute polymer were placed on teflon and glass watch glasses, respectively, and placed in the vacuum oven. The samples were observed at various temperatures and times shown in Figure 6. The maximum temperature was 165°C and the heating cycle lasted 90 minutes. Both samples formed amber, resinous melts after 300 minutes in the oven as shown in Figure 6. Cooled, fused samples of PVP/VA were easily removed from the teflon and readily fractured into 1/4 - 3/8 inch pieces for use in the cryofracturing mill. This material cryofractured easily, permitting 94.2 grams to be blended with 10 gallons of fuel over a 2 to 4 minute blending time at 4 gallons per minute.

Blend Of 75 Percent Concentration Of FM-9 In JET A. Nine<sup>+/-</sup> four point two (94.2) grams of PVP/VA powder was blended with 31.4 grams of JET A fuel forming a heavy slurry. The resulting slurry was placed into a small extruder. The slurry was extruded in drops into a container containing liquid nitrogen. Frozen drops approximately 1/8 inch diameter were formed. The frozen slurry of PVP/VA and JET A was easily run through the cryofracturing mill permitting 125.6 grams to be blended with 10 gallons of JET A in 2 to 4 minutes. The flow rate corresponds to 4 gallons per minute and the total time is 2.5 minutes.

Blending An FM-9 Slurry In JET A. No information was made available regarding the physical properties of the FM-9 slurry. The approach established for the blend of FM-9 in JET A was adopted for the FM-9 slurry.

#### APPLICATION OF THE GTA BLENDING PROCESS TO FM-9.

PELLETIZING BY PRESSURE UNDER VACUUM. FM-9 powder was made available for the preparation of pellets on the morning of January 6, 1981. An attempt was made to produce a 5 gram fused pellet in the press at 10 tons per square inch for 2 to 3 minutes under vacuum at room temperature. The result was a packed powder which could be scraped away readily in powder form with a sharp instrument.

It was decided to determine the minimal heat required to produce a satisfactory 5 gram pellet of FM-9.

Test 1. A 5 gram sample of FM-9 was placed in the 1 1/8 inch dia. cell and subjected to evacuation for 2 minutes and then placed under 10-12 tons per square inch while heating for 3 minutes. The sample was left under pressure for an additional 4 minutes while cooling. There was some evidence of fusion, particularly on the piston side of the pellet. It was decided that the vacuum jacket should be removed to increase the temperature of the pellet during the heating period.

FIGURE 6

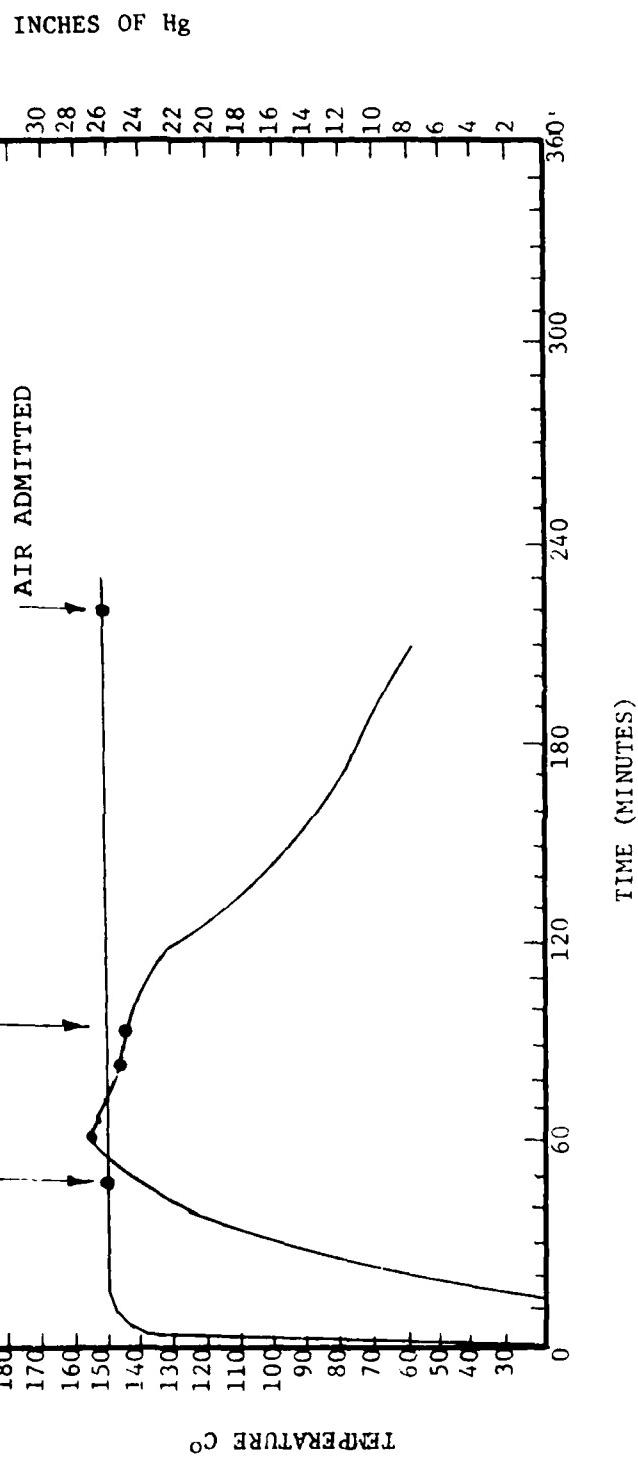
VACUUM OVEN FUSION OF  
PVP/VA SUBSTITUTE POLYMER

OVEN SEALED BUT VACUUM PUMP OFF AT 46 MINUTES

HEAT OFF AT 88 MINUTES

AIR ADMITTED

TEMPERATURE  $^{\circ}$ C



Test 2. A 7 gram sample of FM-9 was placed in the pressure cell and after evacuating for 2 minutes, was subject to the 10-12 tons per square inch pressure while being heated for 5 minutes. A thermometer placed on the outer surface of the cell registered a maximum temperature of 75°C. The cell was allowed to cool under pressure for an additional 10 minutes. The appearance was improved over the result of Test 1. It was difficult to fit the cell under the press piston and it was decided that the sample size be held to less than 7 grams.

Test 3. A 5 gram sample of FM-9 was placed in the pressure cell and the cell was placed in the vacuum jacket. Prior to compression, the sample was evacuated for 3 minutes. The cell and vacuum jacket were placed in the press and subjected to 10-12 tons per square inch and heat for 7 minutes. A temperature of 52°C registered at the surface of the vacuum jacket. After disassembly, a temperature of 53°C registered on the surface of the pressure cell.

Test 4. A 5 gram sample of FM-9 was subjected to 10-12 tons per square inch and heated for 5 minutes. The maximum temperature registered on the outside of the vacuum jacket was 60°C. Heating was continued for an additional 5 minutes. The surface temperature at the end of the second heating period was 70°C. It was decided to apply vacuum to the sample prior to heating under pressure in order to achieve higher pellet temperatures.

Test 5. A 5 gram sample of FM-9 was placed under vacuum at room temperature. The sample was compressed under vacuum and subjected to 10-12 tons per square inch at room temperature for 2 minutes. The vacuum jacket was removed and the pressure cell was subjected to 10-12 tons per square inch while being heated for 5 minutes. The outside temperature of the cell was 78°C.

Test 6. A 5 gram sample of FM-9 was evacuated and compressed as in Test 5 at 10-12 tons per square inch at room temperature. The vacuum jacket was removed as in Test 5 with subsequent heating for 7 minutes at 10-12 tons per square inch. The temperature maximum observed at the outside of cell surface was between 88 and 90°C. The cell was removed after 10 minutes elapsed time and the pellet was removed while hot.

Sufficient pellets were prepared to total 95.5 grams. The pellets were placed in a polyethylene bag and were fractured by hand into approximately 1/8 inch to 1/4 inch pieces. The fragments of pelletized FM-9 were cooled in liquid nitrogen and introduced in GTA's cryofracturing mill at an average rate of approximately 38.2 grams per minute. A mix of 95.5 grams in 10 gallons of JET A was produced in approximately 2.5 minutes.

A sample of FM-9 JET A fuel mix was taken from the spigot of the collecting barrel approximately 30 seconds after completion of the blending operation. At first, the appearance of the FM-9/JET A mix suggested that the FM-9 was going into solution. Within 2 minutes, however, flocculation was observed and shortly thereafter the FM-9

appeared to separate from the JET A. A second sample taken from the collection barrel showed a distinct separation of the FM-9 from the JET A confirming the first observation.

The contents of the collection barrel was drained into two 5 gallon containers. It was then observed that a major portion of the FM-9 had settled at the bottom of the barrel in a slightly swollen form.

GTA learned from the representative of Southwest Research Institute that batch-blending of FM-9 powder without carrier with JET A was accomplished at elevated temperatures. As a result an attempt was made to dissolve cryofractured FM-9 particles in JET A which had been preheated to a temperature of 100°F. Approximately 200 mls of JET A was heated to 100°F on a electric hot plate. The 25 grams of FM-9 particles was cryofractured into a vessel containing liquid nitrogen. Most of the liquid nitrogen was allowed to boil off. A slurry containing approximately 1 gram of FM-9 powder and a small amount of liquid nitrogen was introduced into the heated JET A. There was no evidence that any of the FM-9 went into solution.

MELTING THE FM-9 UNDER VACUUM. Shortly after the arrival of the FM-9 powder on the morning of January 6, 1981 information was received from the FAA that the manufacturer of FM-9 stated that the "FM-9 powder flow point is 100°C." A test run on a heated-stage melting point apparatus indicated that the FM-9 supplied to GTA began to flow at 200°C and melted at 230°C. There was some evidence of decomposition. The melting point test was repeated a second time with an identical result.

A 105 gram sample of FM-9 powder was placed in the teflon tray and placed into the vacuum oven. The temperature was set on maximum. After approximately 1 hour the temperature stabilized near 200°C. There was evidence of yellowing, browning and decomposition of the FM-9. The powder volume reduced as the heating process continued. A liquid collected in the liquid nitrogen trap in the vacuum line. It was determined that this approach could not be used to prepare a fused sample of FM-9. After cooling, inspection of the vacuum trap revealed the presence of a liquid that probably resulted from the decomposition of FM-9.

In a second attempt at fusing FM-9, a 104 gram sample of the polymer was placed in a 500 ml round-bottom flask. The flask was evacuated and heated in a Glas Col heating mantle. Browning was noted as the powder in contact with the surface of the flask appeared to melt. Bubbling was observed in the liquid regions. Over a 1 hour period, the congealed mass revealed colors passing from yellow through black with the darker colors appearing closer to the surface of the flask.

After cooling and removing the mass, condensed liquids were found in the vacuum lines and liquid nitrogen trap. Due to decomposition, a fused sample of FM-9 could not be prepared by heating under vacuum.

BLEND OF 75 PERCENT CONCENTRATION OF FM-9 IN JET A. An attempt to produce a slurry consisting of 75 percent FM-9 powder and 25 percent JET A produced a slightly wetted powder. Increasing amounts of JET A (seven consecutive additions of 34.5 grams of JET A to 107 grams of FM-9) finally produced a fully wetted powder at a composition of 31 percent FM-9 powder and 69 percent JET A. The resulting slurry could not be expelled from the GTA extruder and was subsequently separated by hand into 1/8 to 1/4 inch chunks. These were dropped into liquid nitrogen for freezing prior to grinding.

Three hundred forty-eight (348) grams of the frozen FM-9/JET A particles were cryofractured into 10 gallons of JET A with the intention of producing a 0.3 percent blend of FM-9 in JET A. Immediately upon completion of the blending operation a 200 ml sample was drawn from the spigot of the collection barrel. Visual observation indicated that the FM-9 had not dissolved appreciably in the JET A. Tests carried out by the FAA observer team appeared to reach the same conclusion.

The collection barrel was drained into two 5 gallon cans. A large mass of swollen polymer was found at the bottom of the barrel.

BLENDING FM-9 SLURRY INTO JET A. A can of premixed FM-9 slurry was brought to GTA by the representative of the Southwest Research Institute. It proved to be a wetted powder and not susceptible to extrusion in the GTA extruder. The FM-9 slurry was separated by hand into particles of a size which could be processed by the cryofracturing mill. The particles were then frozen in liquid nitrogen.

The FM-9 slurry is comprised of 32 percent FM-9; the rest being a glycol and amine mixture. Three hundred (300) grams of the slurry were cryofractured in 12 gallons of JET A to produce a 0.3 percent solution of FM-9 in JET A.

A sample of the FM-9 slurry/JET A fuel mix was taken from the collection barrel immediately after the blending operation was completed. The sample was turbid in appearance, indicating that the FM-9 had not gone into solution. In an attempt to enhance solution a drawn sample was placed in an erlenmeyer flask and stirred with a magnetic stirrer. This attempt resulted in a separation of the FM-9 from the JET A solvent.

Samples of the FM-9 slurry/JET A fuel mix drawn from the collection barrel after 15 minutes were less turbid suggesting that the FM-9 was going into solution. Since the FM-9 slurry is designed to go into solution 15 minutes after agitation, an attempt was made to put FM-9 slurry directly into solution in JET A in a beaker. Agitation was provided by a magnetic stirrer during the addition and thereafter. There was no visual evidence that the FM-9 slurry was in solution at or beyond 15 minutes elapsed time.

After 30 minutes, observations were made of the initial sample drawn from the collection barrel immediately after blending. The sample had cleared considerably and was evidently going into solution. Two

hours later this sample still had some turbidity.

#### CONCLUSIONS

1. The GTA blending process produced a blend of Oppanol B-230 and JET A fuel which had antimisting behavior within minutes after the blending operation began.
2. Tests of a 0.139 percent concentration of Oppanol B-230 in JET A in the FAA's Flammability Comparison Test Apparatus indicate flammability suppression roughly equivalent to a 0.3 percent FM-9 fuel with carrier.
3. The GTA blending process was not effective in blending FM-9 powder as supplied by the manufacturer in JET A.
4. The GTA process did not put an FM-9 slurry (with carrier) in solution instantaneously.

#### LIST OF REFERENCES

1. "Engineering and Development Program Plan Antimisting Fuel," Report No. FAA-ED-18-4, June 1980.
2. Mannheimer, R.J., "Restoring Essential Flow and Ignition Properties to Antimisting Kerosene (AMK) For Turbine Aircraft Operations," Report No. FAA-RD-79-62.

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